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Indian Standard

SPECIFICATION FOR METHYL PARATHION EMULSIFIABLE CONCENTRATES

(First Revision)

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Gr 4 + Gr 2 *July* 1978

Indian Standard

SPECIFICATION FOR METHYL PARATHION EMULSIFIABLE CONCENTRATES

(First Revision)

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AMENDMENT NO. 1 NOVEMBER 1980
TO
IS : 2865 - 1978 SPECIFICATION FOR METHYL PARATHION EMULSIFIABLE CONCENTRATES

(First Revision)

Alterations

(*Page* 4, *clause* **2.2.4**) — Substitute the following for the existing clause:

'**2.2.4** *Emulsion Stability* — Any separation, including creaming at the top and sedimentation at the bottom, of 100 ml of emulsion prepared in standard hard water with 2 ml of concentrates shall not exceed 2 ml when tested by one of the methods prescribed in **13.3** of IS : 6940-1973†.'

(*Pages* 10 *to* 13, *clauses* **A-2** *to* **A-2.4**) —Substitute the following for the existing clauses :

'**A-2. TITRATION METHOD**

A-2.1 Principle — Methyl parathion is separated from the most likely impurity *p*-nitrophenol by treating the benzene solution with chilled (10°C) 10 percent (*m/v*) sodium bicarbonate solution. The benzene solution of methyl parathion is reduced by zinc and acetic acid-hydrochloric acid mixture. The amino groups formed are titrated with standard sodium nitrite solution.

A-2.2 Reagents

A-2.2.1 *Benzene*

A-2.2.2 *Sodium Bicarbonate Solution* — 10 percent (*m/v*).

A-2.2.3 *Sodium Sulphate* — anhydrous.

A-2.2.4 *Acetic Acid-Hydrochloric Acid Mixture* — prepared by mixing glacial acetic acid and concentrated hydrochloric acid (9 : 1 *v/v*).

A-2.2.5 *Zinc Dust* — iron free.

A-2.2.6 *Acetic Acid* — glacial.

A-2.2.7 *Hydrochloric Acid* — concentrated.

A-1.2 Apparatus

A-1.2.1 *Volumetric Flasks* — 100-ml, 250-ml, 500-ml and 1 000-ml capacity.

A-1.2.2 *Pipettes* — 20-ml, 25-ml and 50-ml capacity.

A-1.2.3 *Beakers*

A-1.2.4 *Conical Flask* — 250-ml capacity of standard joints.

A-1.2.5 *Separating Funnel* — 250-ml capacity.

A-1.2.6 *UV Visible Spectrophotometer*

A-1.3 Reagents

A-1.3.1 *Methanol*

A-1.3.2 *Diethyl Ether* — (Solvent Ether).

A-1.3.3 *Sodium Hydroxide Solution* — 1N standard.

A-1.3.4 *Sodium Hydroxide Solution* — 0·1N standard.

A-1.3.5 *Sodium Carbonate Solution* — 1 percent (*m*/*m*) chilled.

A-1.3.6 *Methyl Parathion* — of known purity.

A-1.4 Procedure

A-1.4.1 *Preparation of Standard Methyl Parathion Solution*

A-1.4.1.1 Weigh accurately about 0 8 g of pure methyl parathion in 250-ml conical flask. To it add 50-ml methanol and 25-ml 1N standard sodium hydroxide solution. Heat the mixture under reflux condenser for 1 hour. Cool for 15 minutes and wash the condenser with 100-ml of 0·1N standard sodium hydroxide solution followed by 25-ml of distilled water: cool to room temperature and transfer the contents quantitatively to 1 000-ml volumetric flask with distilled water. Shake well to mix the contents. Call it *solution A.*

A-1.4.1.2 Transfer 25-ml of *solution A* into a 250-ml volumetric flask and make up the volume with distilled water. Shake well to get a uniform solution. Call it *solution B.*

A-1.4.1.3 Transfer 10-ml of *solution B* into a 100-ml volumetric flask and make up the volume with distilled water, shake well to get a uniform solution. Call it *solution C.*

A-1.4.1.4 Measure the absorbance of *solution C* at 400 nm against distilled water.

A-1.4.2 *Preparation of Sample Solution*

A-1.4.2.1 Weigh accurately about 1·5 g of sample in 250-ml conical flask and proceed exactly as described in **A-1.4.1**.

A-1.4.3 *Determination of Free p-nitrophenol*

A-1.4.3.1 Weigh accurately about 0·15 g of sample in a 50-ml beaker. Transfer it to a 250-ml separating funnel containing 50-ml solvent ether quantitatively with about 50-ml solvent ether. Extract the free *p*-nitrophenol with 20-ml of 1 percent chilled sodium carbonate solution. Continue the extraction, till the aqueous sodium carbonate phase is colourless. Depending on the volume of carbonate washings transfer it to 100-ml, 250-ml or 500-ml volumetric flask quantitatively and make up the volume with distilled water. Prepare a 1 percent sodium carbonate solution blank by diluting the equivalent volume of 1 percent sodium carbonate solution required for the extraction of *p*-nitrophenol to the same volume as it is done for *p*-nitrophenol extract. Shake well to get uniform solutions. Measure the absorbance of *p*-nitrophenol extract in sample cell and sodium carbonate blank solution in reference cell.

A-1.4.4 *Calculation*

$$\text{Metyhl parathion, percent by mass} = \frac{(A_2 \times M_1 \times P)}{(A_1 \times M_2)} - \frac{(A_3 \times M_1 \times P)}{(A_1 \times M_3 \times F)}$$

where

A_2 = absorbance of sample;

M_1 = mass, in g, of the standard;

P = percent purity of standard methyl parathion;

A_1 = absorbance of standard;

M_2 = mass, in g, of the sample;

A_3 = absorbance of *p*-nitrophenol extract;

M_3 = mass, in g, of sample taken for *p*-nitrophenol extraction; and

F = dilution factor which will be

500 if *p*-nitrophenol extract is diluted to 100-ml
200 if *p*-nitrophenol extract is diluted to 250-ml
100 if *p*-nitrophenol extract is diluted to 500-ml

A-2. TITRATION METHOD

A-2.1 Principle — Methyl parathion is separated from the most likely impurity *p*-nitrophenol by treating the benzene solution with chilled (10°C) 10 percent (*m/v*) sodium bicarbonate solution. The benzene solution of methyl parathion is reduced by zinc and acetic acid-hydrochloric acid mixture. The amino groups formed are titrated with standard sodium nitrite solution.

A-2.2 Reagents

A-2.2.1 *Benzene*

A-2.2.2 *Sodium Bicarbonate Solution* — 10 percent (*m/v*).

A-2.2.3 *Sodium Sulphate* — anhydrous.

A-2.2.4 *Acetic Acid-Hydrochloric Acid Mixture* — prepared by mixing glacial acetic acid and concentrated hydrochloric acid (9:1 *v/v*).

A-2.2.5 *Zinc Dust* — iron free.

A-2.2.6 *Acetic Acid* — glacial.

A-2.2.7 *Hydrochloric Acid* — concentrated.

A-2.2.8 *Sodium or Potassium Bromide*

A-2.2.9 *Sulphanilic Acid* — anhydrous.

A-2.2.10 *Standard Sodium Nitrite Solution* — 1·0 M, prepared by dissolving 6·909 g of sodium nitrite in water and making up to one litre with water. This solution is standardized as given in **A-2.2.10.1**.

A-2.2.10.1 Weigh accurately 0·40 to 0·45 g of anhydrous sulphanilic acid into a 400-ml tall-form beaker. Add 80-ml of water, 10-ml of concentrated hydrochloric acid, 30-ml of glacial acetic acid, and 5 g of sodium or potassium bromide. Cool the mixture to 0° to 10°C by the addition of clean shaved ice and place under mechanical stirring. Titrate this mixture at 0° to 10°C with the 0·1 M sodium nitrite solution as rapidly as the spot test permits. Near the end point, add the sodium nitrite solution in 4-drop portions. Calculate the normality of the 0·1 M sodium nitrite solution as:

$$\text{Normality of the 0·1 M sodium nitrite solution} = \frac{M \times 1\,000}{V \times 173{\cdot}2} = \frac{M \times 5{\cdot}774}{V}$$

where

M = mass, in g, of the sulphanilic acid used, and

V = volume, in ml, of 0·1 M sodium nitrite solution used.

A-2.3 Procedure

A-2.3.1 *Separation and Extraction of Methyl Parathion* — To a 250-ml separatory funnel containing 100-ml benzene and 1 to 2 g of anhydrous sodium sulphate, add accurately weighed sample containing 0·8 to 1·0 g of methyl parathion. Add 25 ml of 10 percent chilled sodium bicarbonate solution and very carefully and slowly extract the free *p*-nitrophenol. Drain out the clear yellow coloured aqueous layer. If any emulsion formation is observed then only clear portion is removed. Add again 1 to 2 g of anhydrous sodium sulphate followed by chilled 10 percent sodium bicarbonate solution and the extraction of the free *p*-nitrophenol is carried out as described above. Continue the extraction with sodium bicarbonate solution till all the *p*-nitrophenol is extracted as indicated by absence of yellow colour in the aqueous phase.

A-2.3.2 *Determination of Methyl Parathion Content* — Transfer the benzene layer to 500-ml round bottom flask quantitatively by washing the separatory funnel a few times with the solvent and transferring the washing to the flask. Remove the solvent by distillation. Add 35 ml of acetic acid-hydrochloric acid mixture and 3 g of zinc dust.

Cover the flask with a funnel and heat on a steam-bath for 30 minutes. Add 25 ml concentrated hydrochloric acid and continue the heating until the zinc is completely dissolved. Cool and transfer the contents of round bottom flask into a tall-form beaker quantitatively by washings with distilled water. Add 5 g of sodium or potassium bromide. Cool to 0° to 10°C by the addition of clean shaved ice and place under mechanical stirring. Titrate the mixture at 0° to 10°C with the standardized 0·1 M sodium nitrite solution as rapidly as the spot test permits. Near the end-point add the sodium nitrite solution in 4-drop portions.

A-2.3.3 *Spot Test* — Dip a glass rod into the solution to be tested and then touch the rod quickly to a piece of potassium iodide starch paper. The end-point is reached when the intense blue-black colour appears with 15-20 seconds and can be obtained repeatedly during a 1-minute period without further addition of sodium nitrite solution.

A-2.4 Calculation

$$\text{Methyl parathion content, percent by mass} = \frac{VN \times 26{\cdot}30 \times f}{M}$$

where

V = volume, in ml, of the 0·1 M sodium nitrite solution required for the test;

N = normality of the 0·1 M sodium nitrite solution;

f = correction factor; and

M = mass, in g, of the material taken for the test.

A-2.4.1 *Correction Factor* — Determine the value of the correction factor f from the formula:

$$f = \frac{T}{A}$$

where

T = theoretical calculated quantity of the 0·1 M sodium nitrite solution to be used in the nitro group determination of 1·00 g of a pure and solid substance containing one nitro group*; and

A = actual quantity of 0·1 M sodium nitrite solution used in the nitro group determination of 1·000 g of pure, recrystallized sample of the same substance, following the above described method and using the same reagents.

NOTE — The correction factor *f* includes the errors arising from the impurities of the reagents, the method itself and the handling of the method in a given laboratory. Its value shall lie within the range of 0·98 to 1·02.

---

*Since methyl parathion is usually not available as a pure material, *f* is evaluated on the basis of another substance containing one nitro group. The suitable material are:

	Melting Range
o-nitrobenzoic acid	146 to 148°C
m-nitrobenzoic acid	140 ,, 142°C
p-nitrobenzoic acid	240 ,, 242·5°C
3-nitrosalicylic acid, monohydrate	124 ,, 126°C
3-nitrosalicylic acid, anhydrous	144 ,, 146°C
5-nitrosalicylic acid	228 ,, 230°C
p-chloronitrobenzene	83 ,, 84°C
p-bromonitrobenzene	126 ,, 127°C

(AFCDC 6)

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AMENDMENT NO. 2 JUNE 1985

TO

IS:2865-1978 SPECIFICATION FOR METHYL PARATHION EMULSIFIABLE CONCENTRATES

(First Revision)

(*Page 4, clause 2.1.1*) - Substitute the following for the existing clause:

'2.1.1 Methyl parathion technical concentrate employed in the manufacture of this material shall conform to IS: 9372-1980*'.

(*Page 4, clause 2.2.2, line 4*) - Substitute 'IS: 6940-1982†' *for* 'IS: 6940-1973†'.

[*Page 4, clause 2.2.4* (*see* Amendment No. 1), *line 4*] - Substitute 'IS: 6940-1982†' *for* 'IS: 6940-1973†'

(*Page 4, clause 2.2.5, line 2*) - Substitute 'IS: 6940-1982†' *for* 'IS: 6940-1973†'.

(*Page 4, foot-notes with* '*' *and* '†' *marks*) - Substitute the following for the existing foot-notes:

'*Specification for methyl parathion, technical concentrate.

†Methods of test for pesticides and their formulations (*first revision*)'.

(*Page 5, clause 2.3.1*) - Substitute the following for the existing clause:

'2.3.1 *Methyl Parathion Content* - When determined by the method prescribed in Appendix A, the observed methyl parathion content, percent (*m/m*), of any of the samples shall not differ from the nominal value

by more than the percent tolerances applied for the declared nominal value as given below:

Nominal Value, Percent	*Tolerance, Percent*	
Up to 9	+ 10 - 5	of the nominal value'
Above 9 and below 50	± 5	
50 and above	+ 5 - 3	

(*Page 5, after clause 2.3.1.1*) - Add a new clause after clause 2.3.1.1:

'2.3.1.2 The average content of all the samples taken shall be not less than the declared nominal value.'

(*Page 5, clause 2.3.2, line 2*) - Substitute 'IS: 6940-1982*' *for* 'IS: 6940-1973*'.

(*Page 5, clause 3.1, line 2*) - Substitute 'IS:8190(Part II)-1980†' *for* 'IS:8190(Part II)-1976†'.

(*Page 5, foot-notes with '*' and '†' marks*) - Substitute the following for the existing foot-notes:

'*Methods of test for pesticides and their formulations (*first revision*).

†Requirements for packing of pesticides: Part II Liquid pesticides (*first revision*)'.

(*Page 6, clause 4.1 with note*) - Substitute the following for the existing matter:

"4.1 Representative samples of the material shall be drawn as prescribed in IS:10627-1983 'Methods for sampling of pesticidal formulations'."

(AFCDC 6)

Printed at Prabhat Offset Press, New Delhi-2

AMENDMENT NO. 3 NOVEMBER 1987
TO
IS : 2865 - 1978 SPECIFICATION FOR METHYL PARATHION EMULSIFIABLE CONCENTRATES

(First Revision)

[*Cover page, Pages* 1 *and* 3, *title* (*see also Amendments No.* 1 *and* 2)]— Substitute 'EC' *for* 'Emulsifiable Concentrates'.

[*Page* 4, *clause* **2.2.4** (*see also Amendment No.* 1)] — Substitute '2·0' *for* '2', wherever occurs.

(*Page* 4, *clause* **2.2.5**) — Add the following note under this clause:

'NOTE — The material need not be put to heat stability tests, if it has crossed half of its shelf life.'

[*Page* 5, *clause* **3.2** (b)] — Substitute the following for the existing item:

'b) Name of the manufacturer and date of expiry.'

[*Pages* 6 *to* 13, *Appendix* A (*see also Amendment No.* 1)] — Substitute the following for the existing appendix:

APPENDIX A

(*Clause* 2.3.1)

DETERMINATION OF METHYL PARATHION CONTENT

A-0. GENERAL

A-0.1 For the determination of methyl parathion content, two methods, namely, UV visible spectrophotometric method (*see* **A-1**) and titration method (*see* **A-2**) have been specified. Either of these may be used for routine testing but UV visible spectrophotometric method will be referee method in case of dispute.

A-1. U.V. VISIBLE SPECTROPHOTOMETRIC METHOD

A-1.1 Principle — Sodium *p*-nitrophenolate is obtained by the alkaline hydrolysis of methyl parathion. The liberated sodium *p*-nitrophenolate is determined colorimetrically at 400 mm by UV visible spectroscopy and is then converted to methyl parathion by calculation. Similarly free *p*-nitrophenol is determined and the corrected methyl parathion content is obtained by calculation.

A-2.2.8 *Sodium or Potassium Bromide.*

A-2.2.9 *Sulphanilic Acid* — anhydrous.

A-2.2.10 *Standard Sodium Nitrite Solution* — 0·1 M, prepared by dissolving 6·909 g of sodium nitrite in water and making up to one litre with water. This solution is standardized as given in **A-2.2.10.1**.

A-2.2.10.1 Weigh accurately 0·40 to 0·45 g of anhydrous sulphanilic acid into a 400-ml tall-form beaker. Add 80 ml of water, 10 ml of concentrated hydrochloric acid, 30 ml of glacial acetic acid, and 5 g of sodium or potassium bromide. Cool the mixture to 0° to 10°C by the addition of clean shaved ice and place under mechanical stirring. Titrate this mixture at 0° to 10°C with the 0·1 M sodium nitrite solution as rapidly as the spot test permits. Near the end point, add the sodium nitrite solution in 4-drop portions. Calculate the normality of the 0·1 M sodium nitrite solution as :

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A-2.4.1 *Correction Factor* — Determine the value of the correction factor f from the formula:

$$f = \frac{T}{A}$$

where

T = theoretical calculated quantity of the 0·1 M sodium nitrite solution to be used in the nitro group determination of 1·00 g of a pure and solid substance containing one nitro group*; and

A = actual quantity of 0·1 M sodium nitrite solution used in the nitro group determination of 1·000 g of pure, recrystallized sample of the same substance, following the above described method and using the same reagents.

NOTE — The correction factor *f* includes the errors arising from the impurities of the reagents, the method itself and the handling of the method in a given laboratory. Its value shall lie within the range of 0·98 to 1·02.

---

* Since methyl parathion is usually not available as a pure material, *f* is evaluated on the basis of another substance containing one nitro group. The suitable materials are:

	Melting Range
o-nitrobenzoic acid	146 to 148°C
m-nitrobenzoic acid	140 ,, 142°C
p-nitrobenzoic acid	240 ,, 242·5°C
3-nitrosalicylic acid, monohydrate	124 ,, 126°C
3-nitrosalicylic acid, anhydrous	144 ,, 146°C
5-nitrosalicylic acid	228 ,, 230°C
p-chloronitrobenzene	83 ,, 84°C
p-bromonitrobenzene	126 ,, 127°C

(AFCDC 6)

Printed at Prabhat Offset Press, New Delhi-2

AMENDMENT NO. 4 SEPTEMBER 1988

TO

IS : 2865 - 1978 SPECIFICATION FOR METHYL PARATHION EMULSIFIABLE CONCENTRATES

(First Revision)

(*Page* 4, *clause* 2.2.5) — Add the following note at the end of the clause:

'NOTE — The material need not be subjected to heat treatment if it has crossed half of its shelf life as ascertained from the date of manufacture and date of expiry declared on the container.'

(AFCDC 6)

Printed at Prabhat Offset Press, New Delhi-2

AMENDMENT NO. 5 JULY 1989
TO
IS : 2865 - 1978 SPECIFICATION FOR METHYL PARATHION EMULSIFIABLE CONCENTRATES

(First Revision)

[*Page* 6, *clause* **A-0.1** (*see also Amendment No.* 3)] — Substitute the following for the existing clause:

'**A-0.1** For the determination of methyl parathion content, two methods, namely, UV visible spectrophotometric method (*see* **A-1**) and titration method (*see* **A-2**) have been specified.'

(AFCDC 6)

Printed at Prabhat Offset Press, New Delhi-2

AMENDMENT NO. 6 MAY 1994
TO
IS 2865 : 1978 SPECIFICATION FOR METHYL PARATHION EMULSIFIABLE CONCENTRATES

(First Revision)

(*Page* 4, *clause* **2.2.5**) — Delete.

(*Page* 6, *clause* **4.1**) — Substitute the following for the existing:

'When freshly manufactured material in bulk quantity is offered for inspection, representative samples of the material shall be drawn and tested as prescribed in IS 10627 : 1983 within 90 days of its manufacture. When the material is offered for inspection after 90 days of its manufacture, sampling shall be done as prescribed in IS 10627 : 1983. However, the criteria for conformity of the material when tested, shall be the limits of tolerances, as applicable over the declared nominal value and given under clause **2.3.1** of the standard.'

(FAD 1)

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Indian Standard

SPECIFICATION FOR METHYL PARATHION EMULSIFIABLE CONCENTRATES

(First Revision)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 20 March 1978, after the draft finalized by the Pest Control Sectional Committee had been approved by the Agricultural and Food Products Division Council and the Chemical Division Council.

0.2 This standard was first issued in 1964. Subsequently six amendments were issued from time to time mainly to bring it up to date in respect of tolerances and packaging. Recently some changes in quality of raw materials, particularly solvents necessitated modification of testing procedure for the active ingredient. Opportunity has also been taken to incorporate spectrophotometric method as an alternate referee method.

0.3 Methyl parathion emulsifiable concentrates are largely used in the control of insect pests and mites of agricultural importance.

0.4 Methyl parathion emulsifiable concentrate is generally manufactured to contain 50 percent (*m*/*m*) of methyl parathion.

0.5 In the preparation of this standard, due consideration has been given to the provisions of the Insecticides Act, 1968 and the Rules framed thereunder. However, this standard is subject to the restrictions imposed under these, wherever applicable.

0.6 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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*Rules for rounding off numerical values (*revised*).

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for methyl parathion emulsifiable concentrates.

2. REQUIREMENTS

2.1 Constituents — The material shall consist of methyl parathion, technical, dissolved in suitable solvent(s), together with emulsifying agent(s), and with or without stabilizer(s).

2.1.1 Methyl parathion, technical, employed in the manufacture of this material shall conform to IS : 2570-1963*.

2.2 Physical — The material shall comply with the physical requirements given in **2.2.1** to **2.2.5**.

2.2.1 *Description* — The material shall be a homogeneous stable liquid, free from sediment. Suspended impurities shall be negligible.

2.2.2 *Cold Test* — No turbidity or separation of solid and/or oily matter shall occur when the material is subjected to the cold test at 10°C as prescribed in **13.1** of IS : 6940-1973† or any other lower temperature as agreed to between the purchaser and the vendor.

2.2.3 *Flash Point* (*Abel*) — When determined by the method prescribed in IS : 1448 [P : 20]-1960‡, the flash point of the material shall be above 24·5°C.

2.2.4 *Emulsion Stability* — Any separation, including creaming at the top and sedimentation at the bottom, of 100 ml of emulsion prepared in standard hard water with 5 ml for public health use and 2 ml for agricultural use, shall not exceed 2 ml when tested by one of the methods prescribed in **13.3** of IS : 6940-1973†.

2.2.5 *Heat Stability* — After treating in accordance with the method prescribed in **13.4** of IS : 6940-1973† the material shall comply with the requirements of **2.2.2** and **2.2.4**.

2.3 Chemical — The material shall comply with the chemical requirements given in **2.3.1** and **2.3.2**.

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*Specification for methyl parathion, technical.

†Methods of tests for pesticides and their formulations.

‡Methods of test for petroleum and its products, P : 20 Flash point by Abel's apparatus.

2.3.1 *Methyl Parathion Content* — When determined by the method prescribed in Appendix A, the observed methyl parathion content, percent (*m/m*), of any of the samples shall not differ from the nominal value by more than the percent tolerances applied to the declared nominal value as given below:

Nominal Value	*Tolerance on the Nominal Value, Percentage*
Up to 9	$\begin{cases} +10 \\ -5 \end{cases}$
10 and below 50	± 5
50 and above	$\begin{cases} +5 \\ -3 \end{cases}$

2.3.1.1 The actual value of the methyl parathion content in the formulation shall be calculated to the second decimal place for rounding off to the first decimal place before applying the tolerances given in **2.3.1**.

2.3.2 *Acidity/Alkalinity* — When tested by the method prescribed in **11.3** of IS : 6940-1973*, the acidity (as H_2SO_4), or alkalinity (as NaOH) of the material shall be not more than 1·0 and 0·2 percent by mass respectively.

3. PACKING AND MARKING

3.1 Packing — The material shall be packed according to requirements given in IS : 8190 (Part II) -1976†.

3.2 Marking — The containers shall bear legibly and indelibly the following information and any other additional information as is necessary under the Insecticides Act and Rules:

a) Name of the material;
b) Name of the manufacturer;
c) Date of manufacture;
d) Batch number;
e) Net volume of contents;
f) Nominal methyl parathion content, percent (*m/m*); and
g) The cautionary notice as worded in the Insecticides Act and Rules.

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*Methods of tests for pesticides and their formulations.

†Requirements for packing of pesticides: Part II Liquid pesticides.

3.2.1 The product may also be marked with Standard Mark.

3.2.2 The use of the Standard Mark is governed by the provisions of the Bureau of Indian Standards Act, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to manufactures or producers may be obtained from the Bureau of Indian Standards.

4. SAMPLING

4.1 Representative samples of the material shall be drawn as prescribed in the 'Indian Standard methods for sampling of pesticides and their formulations' (*under preparation*).

NOTE — Till such time the standard under preparation is published the matter shall be as agreed to between the concerned parties.

5. TESTS

5.1 Tests shall be carried out by the appropriate methods referred to in **2.2.1** to **2.2.5**, **2.3.1** and **2.3.2**.

5.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

APPENDIX A

(*Clause* 2.3.1)

DETERMINATION OF METHYL PARATHION CONTENT

A-0. GENERAL

A-0.1 For the determination of methyl parathion content two methods, namely, spectrophotometric method (*see* **A-1**) and titration method (*see* **A-2**) have been specified. Either one of these methods may be used for routine testing, but the spectrophotometric method shall be used as referee method in case of dispute.

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*Specification for water for general laboratory use (*second revision*).

A-1. SPECTROPHOTOMETRIC METHOD

A-1.1 Principle — After alkaline hydrolysis, the total *p*-nitrophenol is determined spectrophotometrically at 405 nm. The content of free *p*-nitrophenol and that from readily hydrolysed impurities is subtracted from the total *p*-nitrophenol. The methyl parathion content may then be calculated.

A-1.2 Apparatus

A-1.2.1 *Spectrophotometer*

A-1.2.2 *Volumetric Flask* — 100-ml capacity.

A-1.2.3 *Measuring Cylinder* — 100-ml capacity.

A-1.2.4 *Reflux Condenser* — Standard joint.

A-1.3 Reagents

A-1.3.1 *Ethanol* — 96 percent.

A-1.3.2 *Aqueous Ethanol* — 50 percent (*v/v*).

A-1.3.3 *Isopropanol*

A-1.3.4 *Sodium Hydroxide* — 1 N.

A-1.3.5 *Ethanolic Sodium Hydroxide* — 1 N.

A-1.3.6 *p-Nitrophenol* — purified (*mp* 114°C).

A-1.4 Procedure

A-1.4.1 *Determination of Total p-Nitrophenol* — Weigh accurately about 50 mg of methyl parathion and transfer to the hydrolysis flask. Add 10 ml of isopropanol and 10 ml of sodium hydroxide (*see* **A-1.3.4**). Keep the mixture at room temperature for 24 h. Dilute to volume with aqueous ethanol (*see* **A-1.3.2**), stopper and shake.

A-1.4.1.1 Pipette 10 ml of solution (*see* **A-1.4.1**) into a second 100-ml volumetric flask, add 10 ml ethanolic sodium hydroxide (*see* **A-1.3.5**), dilute to volume with aqueous ethanol, stopper and shake.

A-1.4.1.2 Pipette 10 ml of solution (*see* **A-1.4.1.1**) into a third 100-ml volumetric flask, add 10 ml ethanolic sodium hydroxide, dilute to volume with aqueous ethanol, stopper and shake. This solution contains *p*-nitrophenol equivalent to 0·5 mg of methyl parathion in 50 percent aqueous ethanol and is 0·1 N with respect to sodium hydroxide.

A-1.4.1.3 Measure its absorbance (*C*) or 405 nm in 1 cm cell against 50 percent aqueous ethanol as a blank.

A-1.4.2 *Molar Absorption of p-Nitrophenol in Sodium Hydroxide* — Weigh accurately, about 200 mg of *p*-nitrophenol, transfer to 100 ml volumetric flask, dissolve the *p*-nitrophenol in isopropanol, dilute to volume with isopropanol, and shake.

A-1.4.2.1 Dilute by the method above to give 1 000 fold dilution.

A-1.4.2.2 Determine the absorbance (D) of the dilute solution at 405 nm in a 1 cm cell against aqueous ethanol (*see* **A-1.3.2**) as a blank:

$$\text{Molar absorption } (M) = \frac{139{\cdot}1 \times 1\,000 \times D}{10 \times 0{\cdot}001 \times q} = \frac{13\,910\,000\,D}{q}$$

where

q = mass of *p*-nitrophenol in g.

$$\text{Methyl parathion content based on total } p\text{-nitrophenol, percent by mass } (T) = \frac{C \times 263{\cdot}2 \times 10 \times 100}{M \times m} = \frac{263\,200\,C}{M \times m}$$

where

M = molar absorption, and

m = mass of sample, in g, taken for the test (*see* **A-1.4.1**).

A-1.5 Determination of Free *p*-Nitrophenol and *p*-Nitrophenol from Easily Hydrolysed Impurities

A-1.5.1 *Principle* — The free *p*-nitrophenol and *p*-nitrophenol (from easily hydrolysed impurities) are determined, after gentle hydrolysis in a buffer solution of *p*H 10·3 and calculated as methyl parathion.

A-1.5.2 *Apparatus*

A-1.5.2.1 *Volumetric flask* — 100-ml and 500-ml capacity.

A-1.5.2.2 *Measuring cylinder* — 100-ml capacity.

A-1.5.2.3 *Spectrophotometer*

A-1.5.2.4 *p*H *meter*

A-1.5.3 *Reagents*

A-1.5.3.1 *Ethanol*

A-1.5.3.2 *Benzene-isopropanol mixture* — 1 : 9 (*v/v*).

A-1.5.3.3 *Buffer solution* — *p*H 10·3 and 11·2.

a) Mix the buffer solution [prepared as in (b)] (250 ml) with benzene isopropanol mixture (*see* **A-1.5.3.2**) (750 ml). Allow to stand 24 hours. Filter and check its *p*H 10·3.

b) Dissolve anhydrous dimethyl amine (20 g) in water (1 000 ml) and add hydrochloric acid (100 ml of 2 N solution) to give *p*H 11·2.

A-1.5.3.4 *p-nitrophenol* — purified (*mp* 114°C).

A-1.5.4 *Procedure*

A-1.5.4.1 *Preparation of sample* — Weigh accurately, sufficient sample to give a usable absorbance value on the spectrophotometer. Transfer to a 100-ml volumetric flask and dissolve in 10 ml of benzene-isopropanol mixture.

A-1.5.4.2 *Measurement* — Dilute the solution (*see* **A-1.5.4.1**) to volume with the buffer mixture (*p*H 10·3) and shake. Measure the absorbance (G) against the buffer mixture within 2 min to give a value for the content of free *p*-nitrophenol. The absorbance is proportional to the concentration of *p*-nitrophenol. Measure the absorbance (H) against the buffer mixture after 24 h at room temperature to give a value for the content of easily hydrolysed impurities which yield *p*-nitrophenol.

A-1.5.4.3 *Molar absorbance of p-nitrophenol in the buffer mixture* — Weigh, accurately, into each of several 100 ml volumetric flasks a progressive series of amounts of *p*-nitrophenol from 200 to 300 mg (X mg). Dissolve the *p*-nitrophenol in benzene-isopropanol mixture, dilute to volume with the same solution, and shake. Pipette a 10 ml aliquot into another 100-ml volumetric flask, dilute to volume with the benzene-isopropanol mixture and shake. Repeat this dilution once more and finally pipette a 10 ml aliquot into a fourth 100-ml volumetric flask, dilute to volume with the buffer mixture (*p*H 10·3) and shake. Determine the absorbance (J) of the final dilution at 405 nm in a 1 cm cell against the buffer mixture as a blank. Calculate the molar absorbance (M) for each of the *p*-nitrophenol weighings as follows:

$$M = \frac{139\,100 \times J \times 100}{X}$$

Finally, calculate the average value and use it for the same cells in future determinations. Check the value from time to time.

A-1.5.4.4 *Calculations*

Percentage of free *p*-nitrophenol and *p*-nitrophenol from easily hydrolysed impurities, percent by mass $= \dfrac{1\,391\,H}{M \times m}$

Free *p*-nitrophenol expressed as methyl parathion $= \dfrac{G \times 263{\cdot}2 \times 100}{M \times m \times 10}$

Free *p*-nitrophenol and *p*-nitrophenol from easily hydrolysed impurities calculated as methyl parathion, percent by mass $= \dfrac{H \times 263{\cdot}2 \times 100}{M \times m \times 10}$

$$= \frac{2\,632\,H}{M \times m} = Q$$

Methyl parathion content, percent by mass $= T - Q$.

where

H = absorbance of solution (*see* **A-1.5.4.2**);
G = absorbance of solution (*see* **A-1.5.4.2**);
M = molar absorbance (*see* **A-1.5.4.3**);
m = mass of sample, taken for the test, in g; and
T = methyl parathion content based on that *p*-nitrophenol (*see* **A-1.4.2.2**).

NOTE 1 — All pipettes and volumetric flasks must be carefully checked before use. The deviations from the stated values must not be greater than 0·05 ml for 100 ml flasks and 0·015 ml for 10 ml pipettes.

NOTE 2 — If the sample is very impure, carry out one or more intermediate dilutions with the benzene-isopropanol mixture, and finally pipette a 10 ml portion into the 100-ml volumetric flask.

NOTE 3 — Take into account any other preliminary dilutions when calculating the final figures.

A-2. TITRATION METHOD

A-2.1 Principle – Methyl parathion is separated from the emulsifying agent and from *p*-nitrophenol, the most likely impurity, by treating the material with mixed solvent and aqueous ethanol, followed by extraction with sodium bicarbonate solution. The methyl parathion in the mixed solvent layer is reduced by zinc and an acetic acid-hydrochloric acid mixture, and the amino groups formed are titrated with the standard sodium nitrite solution.

A-2.2 Reagents

A-2.2.1 *Aqueous Ethanol* — 50 percent (*v/v*).

A-2.2.2 *Mixed Solvent* — prepared by mixing light petroleum (*bp* 40 to 60°C) and benzene in 60 : 40 (*v/v*).

A-2.2.3 *Sodium Chloride Solution* — saturated.

A-2.2.4 *Sodium Bicarbonate Solution* — 10 percent (*m/v*).

A-2.2.5 *Acetic Acid-hydrochloric Acid Mixture* — prepared by mixing glacial acetic acid and concentrated hydrochloric acid (9 : 1 *v/v*).

A-2.2.6 *Zinc Dust* — iron-free.

A-2.2.7 *Concentrated Hydrochloric Acid*

A-2.2.8 *Acetic Acid* — glacial.

A-2.2.9 *Sodium or Potassium Bromide*

A-2.2.10 *Sulfanilic Acid* — anhydrous.

A-2.2.11 *Standard Sodium Nitrite Solution* — 0·1 M, prepared by dissolving 6 90 g of sodium nitrite in water and making up to one litre with water. This solution is standardized as given in **A-2.2.11.1**.

A-2.2.11.1 Weigh accurately 0·40 to 0·45 g of anhydrous sulfanilic acid into a 400-ml tall-form beaker. Add 80 ml of water, 10 ml of concentrated hydrochloric acid, 30 ml of glacial acetic acid, and 5 g of sodium or potassium bromide. Cool the mixture to 0 to 10°C by the addition of clean, shaved ice and place under mechanical stirring. Titrate this mixture at 0 to 10°C with the 0·1 M sodium nitrite solution as rapidly as the spot test permits. Near the end point, add the sodium nitrite solution in 4-drop portions. Calculate the normality of the 0·1 M sodium nitrite solution as:

$$\text{Normality of the 0·1 M sodium nitrite solution} = \frac{M \times 1\,000}{V \times 173{\cdot}2}$$

$$= \frac{M \times 5{\cdot}774}{V}$$

where

M = mass, in g, of the sulfanilic acid used, and

V = volume, in ml, of 0·1 M sodium nitrite solution required.

A-2.3 Procedure

A-2.3.1 *Separation and Extraction of Methyl Parathion* — Weigh accurately an amount of material containing 0·5 to 0·6 g of methyl parathion and rinse with 100 ml of aqueous ethanol and 100 ml of mixed solvent into a 250-ml separatory funnel. Add 10 ml of saturated sodium chloride solution and shake for 30 seconds. Drain the aqueous ethanol layer in to a second separatory funnel and extract with 50 ml of mixed solvent as above. Repeat this operation at least two times with 50 ml of mixed solvent as above and discard the aqueous ethanol layer. Wash the organic layers:

a) twice with 30 ml portions of aqueous ethanol,
b) twice with 30 ml portions of chilled (10°C) sodium bicarbonate solution, and
c) finally with 50 ml of water to which 10 ml of saturated sodium chloride solution has been added.

A-2.3.2 *Determination of Methyl Parathion in Organic Layer* — Combine the solvent extracts in 500-ml round bottom flask and distil solvent. Add 35 ml of acetic acid-hydrochloric acid mixture and 3 g of zinc dust. Cover the flask with a funnel and heat on a steam bath for at least 30 minutes. Add 10 ml of concentrated hydrochloric acid and continue the heating until the zinc is completely dissolved. Cool, and transfer the contents of round bottom flask into a tall form beaker quantitatively by washing with distilled water. Add 5 g of sodium or potassium bromide. Cool to 0 to 10°C by the addition of clean shaved ice and place under mechanical stirring. Titrate the mixture at 0 to 10°C with the standardized 0·1 M sodium nitrite solution as rapidly as the spot test permits. Near the end point add the sodium nitrite solution in 4-drop portions.

A-2.3.3 *Spot Test* — Dip a glass rod into the solution to be tested and then touch the rod quickly to a piece of potassium iodide starch paper. The end-point is reached when the intense blue-black colour appears within 15-20 seconds and can be obtained repeatedly during a 1-minute period without further addition of sodium nitrite solution.

A-2.4 Calculation

$$\text{Methyl parathion content, percent by mass} = \frac{VN \times 26{\cdot}3 \times f}{M}$$

where

V = volume, in ml, of the 0·1 M sodium nitrite solution required for the test;

N = normality of the 0·1 M sodium nitrite solution;

f = correction factor; and

M = mass, in g, of the material taken for the test.

A-2.4.1.1 *Correction factor* — Determine the value of the correction factor f from the formula:

$$f = \frac{T}{A}$$

where

T = theoretical calculated quantity of the 0·1 M sodium nitrite solution to be used in the nitro group determination of 1·00 g of a pure and solid substance containing one nitro group*; and

A = actual quantity of 0·1 M sodium nitrite solution used in the nitro group determination of 1·000 g of pure, recrystallized sample of the same substance, following the above described method and using the same reagents.

NOTE — The correction factor f includes the errors arising from the impurities of the reagents, the method itself and the handling of the method in a given laboratory. Its value shall lie within the range of 0·98 to 1·02.

---

*Since methyl parathion is usually not available as a pure material, f is evaluated on the basis of another substance containing one nitro group. The suitable materials are:

	Melting Range
o-nitrobenzoic acid	146 to 148°C
m-nitrobenzoic acid	140 ,, 142°C
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5-nitrosalicylic acid	228 ,, 230°C
p-chloronitrobenzene	83 ,, 84°C
p-bromonitrobenzene	126 ,, 127°C

(*Continued from page* 2)

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BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110002
Telephones: 23230131, 23233375, 23239402 **Fax**: 91+011 23239399, 23239382
E - Mail : info@bis.org.in website : http://www.bis.org.in

Central Laboratory:	***Telephone***
Plot No. 20/9, Site IV, Sahibabad Industrial Area, SAHIBABAD 201010	277 0032

Regional Offices:

Central	: Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110002	2323 7617
*Eastern	: 1/14 CIT Scheme VII M, V.I.P. Road, Kankurgachi, KOLKATA 700054	2337 8662
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SCO 21, Sector 12, Faridabad 121007	229 2175
Savitri Complex, 116 G.T. Road, GHAZIABAD 201001	286 1498
53/5 Ward No. 29, R.G. Barua Road, 5th By-lane, Apurba Sinha Path, GUWAHATI 781003	245 6508
5-8-56C, L.N. Gupta Marg, Nampally Station Road, HYDERABAD 500001	2320 1084
Prithavi Raj Road, Opposite Bharat Overseas Bank, C-Scheme, JAIPUR 302001	222 3282
11/418 B, Sarvodaya Nagar, KANPUR 208005	223 3012
Sethi Bhawan, 2nd Floor, Behind Leela Cinema, Naval Kishore Road, LUCKNOW 226001	261 8923
H. No. 15, Sector-3, PARWANOO, Distt. Solan (H.P.) 173220	235 436
Plot No A-20-21, Institutional Area, Sector 62, Goutam Budh Nagar, NOIDA 201307	240 2206
Patliputra Industrial Estate, PATNA 800013	226 2808
Plot Nos. 657-660, Market Yard, Gultkdi, PUNE 411037	2427 4804
"Sahajanand House" 3rd Floor, Bhaktinagar Circle, 80 Feet Road, RAJKOT 360002	237 8251
T.C. No. 2/275 (1 & 2), Near Food Corporation of India, Kesavadasapuram-Ulloor Road, Kesavadasapuram, THIRUVANANTHAPURAM 695004	255 7914
1st Floor, Udyog Bhavan, VUDA, Siripuram Junction, VISHAKHAPATNAM-03	271 2833

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*Sales Office is at 5 Chowringhee Approach, P.O. Princep Street, KOLKATA 700072 2355 3243

†Sales Office (WRO) Plot No. E-9, MIDC, Rd No. 8, Behind Telephone Exchange, Andheri (East), Mumbai-400 0093 2832 9295

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